

Certificate of Analysis

Standard Reference Material® 1941b

Organics in Marine Sediment

Standard Reference Material (SRM) 1941b is marine sediment collected at the mouth of the Baltimore Harbor. SRM 1941b is intended for use in evaluating analytical methods for the determination of selected polycyclic aromatic hydrocarbons (PAHs), polychlorinated biphenyl (PCB) congeners, and chlorinated pesticides in marine sediment and similar matrices. Information is also provided for total organic carbon, carbon, hydrogen, and nitrogen. All of the constituents for which certified, reference, and information values are provided in SRM 1941b were naturally present in the sediment material before processing. A unit of SRM 1941b consists of a bottle containing 50 g of radiation-sterilized, freeze-dried sediment material.

Certified Concentration Values: Certified values for concentrations, expressed as mass fractions, for 24 PAHs, 29 PCB congeners, and 7 chlorinated pesticides are provided in Tables 1 through 3. The certified values for the PAHs, PCB congeners, and chlorinated pesticides are based on the agreement of results obtained at NIST from two or more chemically independent analytical techniques along with results from an interlaboratory comparison study [1,2]. A NIST certified value is a value for which NIST has the highest confidence in its accuracy in that all known or suspected sources of bias have been investigated or accounted for by NIST.

Reference Concentration Values: Reference values for concentrations, expressed as mass fractions, are provided for 44 additional PAHs (some in combination), 13 additional PCB congeners, and 2 additional chlorinated pesticides in Tables 4 to 7. A reference value for total organic carbon is provided in Table 8. Reference values are noncertified values that are the best estimate of the true value. However, the values do not meet the NIST criteria for certification and are provided with associated uncertainties that may reflect only measurement precision, may not include all sources of uncertainty, or may reflect a lack of sufficient statistical agreement among multiple analytical methods.

Information Concentration Values: Information values for concentrations, expressed as mass fractions, are provided in Table 9 for carbon, hydrogen, and nitrogen. An information value is considered to be a value that will be of interest and use to the SRM user, but insufficient information is available to assess adequately the uncertainty associated with the value or only a limited number of analyses were performed.

Expiration of Certification: The certification of this SRM is valid until **01 March 2012**, within the measurement uncertainties specified, provided the SRM is handled and stored in accordance with the instructions given in this certificate. However, the certification is invalid if the SRM is damaged, contaminated, or modified.

Maintenance of SRM Certification: NIST will monitor this SRM over the period of its certification. If substantive technical changes occur that affect the certification before the expiration of this certificate, NIST will notify the purchaser. Registration (see attached sheet) will facilitate notification.

The coordination of the technical measurements leading to the certification of this material was under the leadership of M.M. Schantz and S.A. Wise of the NIST Analytical Chemistry Division.

Willie E. May, Chief Analytical Chemistry Division

Robert L. Watters, Jr., Acting Chief Measurement Services Division

Gaithersburg, MD 20899 Certificate Issue Date: 16 August 2004 See Certificate Revision History on Page 13

SRM 1941b Page 1 of 14

Analytical measurements for the certification of SRM 1941b were performed at NIST by J.R. Kucklick, B.J. Porter, D.L. Poster, M.M. Schantz, P. Schubert, S. Tutschku, and L.L. Yu of the NIST Analytical Chemistry Division. Measurements for percent total organic carbon were provided by a commercial laboratory and T.L. Wade of the Geochemical and Environmental Research Group, Texas A&M University (College Station, TX). The carbon, hydrogen, and nitrogen data were provided by a commercial laboratory. Results were also used from 38 laboratories (see Appendix A) that participated in an interlaboratory comparison exercise coordinated by NIST.

Collection and preparation of SRM 1941b were performed by M.P. Cronise and C.N. Fales of the NIST Measurement Services Division and B.J. Porter and M.M. Schantz of the NIST Analytical Chemistry Division. The sediment material was collected with the assistance of G.G. Lauenstein, J. Collier, and J. Lewis (National Oceanic and Atmospheric Administration).

Consultation on the statistical design of the experimental work and evaluation of the data were provided by S.D. Leigh and J.H. Yen of the NIST Statistical Engineering Division.

The support aspects involved in the issuance of this SRM were coordinated through the NIST Standard Reference Materials Program by B.S. MacDonald of the NIST Measurement Services Division.

NOTICE AND WARNING TO USERS

Storage: SRM 1941b must be stored in its original bottle at temperatures less than 30 °C away from direct sunlight.

Handling: This material is naturally occurring marine sediment from an urban area and may contain constituents of unknown toxicities; therefore, caution and care should be exercised during its handling and use.

INSTRUCTIONS FOR USE

Prior to removal of subsamples for analysis, the contents of the bottle should be mixed. The concentrations of constituents in SRM 1941b are reported on a dry-mass basis. The SRM, as received, contains approximately 2.4 % moisture. The sediment sample should be dried to a constant mass before weighing for analysis; or a separate subsample of the sediment should be removed from the bottle at the time of analysis and dried to determine the concentration on a dry-mass basis. If the constituents of interest are volatile, then the moisture must be determined with a separate subsample.

PREPARATION AND ANALYSIS¹

Sample Collection and Preparation: The sediment used to prepare this SRM was collected from the Chesapeake Bay at the mouth of the Baltimore (MD) Harbor near the Francis Scott Key Bridge (39°12.3'N and 76°31.4'W). This location is very near the site where SRM 1941 and SRM 1941a were collected. The sediment was collected using a Kynar-coated modified Van Veen-type grab sampler. A total of approximately 3300 kg of wet sediment was collected from the site. The sediment was freeze-dried, sieved at 150 μ m (100 % passing), homogenized in a cone blender, radiation sterilized (60 Co), and then packaged in screw-capped amber glass bottles each containing approximately 50 g.

Conversion to Dry-Mass Basis: The results for the constituents in SRM 1941b are reported on a dry-mass basis; however, the material "as received" contains residual moisture. The amount of moisture in SRM 1941b was determined by measuring the mass loss after freeze drying subsamples of 1.1 g to 1.3 g for four days at 1 Pa with a -10 °C shelf temperature and a -50 °C condenser temperature. The moisture content in SRM 1941b at the time of the certification analyses was 2.39 % \pm 0.08 % (95 % confidence level). Analytical results for the organic constituents were determined on an as-received basis and then converted to a dry-mass basis by dividing by the conversion factor of 0.9761 (g dry mass/g as-received mass).

Polycyclic Aromatic Hydrocarbons: The general approach used for the value assignment of the PAHs in SRM 1941b was similar to that reported for the recent certification of several environmental matrix SRMs [3-6] and is described in detail elsewhere [7]. The approach consisted of combining results from analyses using various combinations of different extraction techniques and solvents, clean-up/isolation procedures, and chromatographic separation and detection

SRM 1941b Page 2 of 14

¹Certain commercial equipment, instruments, or materials are identified in this certificate in order to adequately specify the experimental procedure. Such identification does not imply recommendation or endorsement by the National Institute of Standards and Technology, nor does it imply that the materials or equipment identified are necessarily the best available for the purpose.

techniques. This approach consisted of Soxhlet extraction and pressurized-fluid extraction (PFE) using dichloromethane (DCM) or a hexane/acetone mixture, cleanup of the extracts using solid-phase extraction (SPE) or normal-phase liquid chromatography (LC), followed by analysis using the following techniques: (1) reversed-phase liquid chromatography with fluorescence detection (LC-FL) analysis of the total PAH fraction, (2) reversed-phase LC-FL analysis of isomeric PAH fractions isolated by normal-phase LC (i.e., multidimensional LC), (3) gas chromatography/mass spectrometry (GC/MS) analysis of the PAH fraction on three stationary phases of different selectivity, i.e., a 5 % (all column compositions are given as % mole fraction) phenyl-substituted methylpolysiloxane phase, a 50 % phenyl-substituted methylpolysiloxane phase, and a relatively non-polar proprietary phase.

Three sets of GC/MS results, designated as GC/MS (I), GC/MS (II), and GC/MS (III) were obtained using three columns with different selectivities for the separation of PAHs. For GC/MS (I) analyses, duplicate subsamples of approximately 1 g from ten bottles of SRM 1941b were extracted using PFE with DCM [8]. Copper powder was added to the extract to remove elemental sulfur. The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 2 % DCM in hexane (all solvent concentrations are given as % volume fraction). The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-5 MS, J&W Scientific, Folsom, CA). The GC/MS (II) analyses were performed using 5 g subsamples from six bottles of SRM 1941b. These samples were extracted using PFE with DCM. The high molecular mass compounds were removed from the extracts using size exclusion chromatography (SEC) with a preparative-scale divinylbenzene-polystyrene column (10 µm particle size with 100 Å diameter pores), and the sulfur was removed from the extracts by adding copper powder. The concentrated extract was passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The analysis was by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a 50 % phenyl-substituted methylpolysiloxane phase (0.25 µm film thickness) (DB-17 MS, J&W Scientific, Folsom, CA). For the GC/MS (III), 9 g subsamples from six bottles of SRM 1941b were Soxhlet extracted for 18 h with 250 mL of a mixture of 50 % hexane/50 % acetone. Copper powder was added to the extract to remove elemental sulfur, and the concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase (0.25 µm film thickness) (DB-XLB, J&W Scientific, Folsom, CA).

Two sets of LC-FL results, designated as LC-FL (total) and LC-FL (isomer), were used in the certification process. For the LC-FL (total), subsamples of approximately 1 g from six bottles of SRM 1941b were extracted using PFE with a mixture of 50 % hexane/50 % acetone. The extracts were concentrated and then processed through an aminopropylsilane SPE cartridge using 2 % DCM in hexane to obtain the total PAH fraction. For the LC-FL (isomer), a 5 g subsample from the six bottles was extracted using PFE with DCM and processed through an aminopropylsilane SPE cartridge using 10 % DCM in hexane; the PAH fraction was then fractionated further on a semi-preparative aminopropylsilane column (μ Bondapak NH₂, 9 mm i.d. × 30 cm, Waters Associates, Milford, MA) to isolate isomeric PAH fractions as described previously [9-12]. The total PAH fraction and the isomeric PAH fractions were analyzed using a 5 μ m particle-size polymeric octadecylsilane (C_{18}) column (4.6 mm i.d. × 25 cm, Hypersil-PAH, Keystone Scientific, Inc., Bellefonte, PA) with wavelength programmed fluorescence detection [10,11].

For the GC/MS and LC-FL measurements described above, selected perdeuterated PAHs were added to the sediment prior to solvent extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1941b was used in an interlaboratory comparison exercise in 1999 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [13]. Results from 38 laboratories that participated in this exercise were used as the sixth data set in the determination of the certified values for PAHs in SRM 1941b. The laboratories participating in this exercise employed the analytical procedures routinely used in their laboratories to measure PAHs.

Homogeneity Assessment for PAHs: The homogeneity of SRM 1941b was assessed by analyzing duplicate samples of approximately 1 g from ten bottles selected by stratified random sampling. Samples were extracted, processed, and analyzed as described above for GC/MS (I). No statistically significant differences among bottles were observed for the PAHs at this sample size.

PAH Isomers of Molecular Mass 300 and 302: For the determination of the molecular mass 300 and 302 isomers, three subsamples of approximately 5 g each were extracted using PFE with DCM. The extracts were then concentrated with a solvent change to hexane and passed through an aminopropyl SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS using a 0.25 mm i.d. \times 60 m fused silica capillary column with a 50 % phenyl-substitued methylpolysiloxane phase (0.25 μ m film thickness) (DB-17MS, J&W Scientific, Folsom, CA). Perdeuterated dibenzo[a,i]pyrene was added to the sediment prior to extraction for use as an internal standard [14].

SRM 1941b Page 3 of 14

PCBs and Chlorinated Pesticides: The general approach used for the determination of PCBs and chlorinated pesticides in SRM 1941b was similar to that reported for the recent certification of several environmental matrix SRMs [4,5,15-17], and consisted of combining results from analyses using various combinations of different extraction techniques and solvents, cleanup/isolation procedures, and chromatographic separation and detection techniques. This approach consisted of Soxhlet extraction and PFE using DCM or a hexane/acetone mixture, clean-up/isolation using SPE or LC, followed by analysis using GC/MS and gas chromatography with electron capture detection (GC-ECD) on two columns with different selectivity for the separation of PCBs and chlorinated pesticides. The analytical methods are described in detail elsewhere [7].

Six sets of results were obtained designated as GC-ECD (I) A and B, GC/MS (I) A and B, GC/MS (II), and Interlaboratory Comparison Exercise. For the GC-ECD (I) analyses, approximately 10 g subsamples from six bottles of SRM 1941b were extracted using PFE with DCM. Copper powder was added to the extract to remove elemental sulfur, and SEC, as described above, was used to remove the high molecular mass compounds. The concentrated extract was then fractionated on a semi-preparative aminopropylsilane column to isolate two fractions containing: (1) the PCBs and lower polarity pesticides, and (2) the more polar pesticides. GC-ECD analyses of the two fractions were performed on two columns of different selectivities for PCB separations: 0.25 mm \times 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase, (0.25 μ m film thickness, DB-5, J&W Scientific, Folsom, CA), and a 0.25 mm \times 60 m fused silica capillary column with a non-polar proprietary phase, (0.25 μ m film thickness, DB-XLB, J&W Scientific, Folsom, CA). The results from the 5 % phenyl phase are designated as GC-ECD (IA) and the results from the proprietary phase are designated as GC-ECD (IB). For the GC-ECD analyses, two PCB congeners that are not significantly present in the sediment extract (PCB 103 and PCB 198 [18,19]), and endosulfan I- d_4 , d_4 -DDE- d_8 , d_4 -DD- d_8 , and d_4 -DDT- d_8 were added to the sediment prior to extraction for use as internal standards for quantification purposes.

Two sets of results were obtained by GC/MS. For GC/MS (I), approximately 9 g subsamples from six bottles were Soxhlet extracted with a mixture of 50 % hexane/50 % acetone for approximately 18 h. Copper powder was added to the extract to remove elemental sulfur, and the concentrated extract was passed through a silica SPE cartridge and eluted with 10 % DCM in hexane. The processed extract was then analyzed by GC/MS with two ionization modes, electron impact (EI) and negative ion chemical ionization (NICI). The GC/MS EI method, GC/MS (IA), used a 0.25 mm i.d. × 60 m fused silica capillary column with a relatively non-polar proprietary phase, (0.25 μ m film thickness, DB-XLB, J&W Scientific, Folsom, CA). The GC/MS NICI method, GC/MS (IB), used a 0.25 mm i.d. × 60 m fused silica capillary column with a 5 % phenyl-substituted methylpolysiloxane phase, (0.25 μ m film thickness, DB-5MS, J&W Scientific, Folsom, CA). The GC/MS (II) results were obtained in the same manner as the GC/MS (IA) analyses except that three subsamples were Soxhlet extracted with DCM for approximately 18 h. For the GC/MS analyses, selected carbon-13 labeled PCB congeners and chlorinated pesticides were added to the sediment prior to extraction for use as internal standards for quantification purposes.

In addition to the analyses performed at NIST, SRM 1941b was used in an interlaboratory comparison exercise in 1999 as part of the NIST Intercomparison Exercise Program for Organic Contaminants in the Marine Environment [13]. Results from 38 laboratories that participated in this exercise were used as the sixth data set in the determination of the certified values for PCB congeners and chlorinated pesticides in SRM 1941b. The laboratories participating in this exercise employed the analytical procedures routinely used in their laboratories to measure PCB congeners and chlorinated pesticides.

The reference value for PCB 77 was determined from a separate fraction. The samples were extracted and processed as for GC-ECD (I) above. The first (PCB and lower polarity pesticide) fraction from the semi-preparative aminopropylsilane column was further fractionated using a Cosmosil PYE column (5 μ m particle size, 4.6 mm i.d. × 25 cm, Phenomenex, Torrance, CA) [20]. Three fractions were collected: the first fraction contained the pesticides and multi-*ortho* PCBs, the second fraction contained the polychlorinated naphthalenes, non-*ortho* PCB congeners, and some mono-*ortho* PCB congeners, and the third fraction removed the residual planar compounds from the column. The second fraction was analyzed by GC/MS NICI using the same column as GC/MS (IB) above. Carbon-13 labeled PCB 77 was used as an internal standard for quantification purposes.

SRM 1941b Page 4 of 14

Total Organic Carbon: Two laboratories provided results for Total Organic Carbon (TOC) using similar procedures. Briefly, subsamples of approximately 200 mg were reacted with 6 N hydrochloric acid and rinsed with deionized water prior to combustion in a gas fusion furnace. The carbon monoxide and carbon dioxide produced were measured and compared to a blank for calculation of the percent TOC. Each laboratory analyzed subsamples from three bottles of SRM 1941b. One of the laboratories also analyzed three subsamples from three bottles of SRM 1941b for carbon, hydrogen, and nitrogen.

Table 1. Certified Concentrations for Selected PAHs in SRM 1941b

PAHs	Mass Fractions (dry-mass basis) ^a μg/kg		
	•	0 0	5
Naphthalene ^{d,e,f,g,h,i}	848	±	95 ^b
Fluorene ^{d,e,f,g,h,i}	85	±	15 ^b
Phenanthrene ^{d,e,f,g,h,i}	406	±	44 ^b
Anthracene ^{d,e,f,g,h,i}	184	±	18 ^b
3-Methylphenanthrene ^{d,e,f}	105	±	13 ^b
2-Methylphenathrene ^{d,e,f}	128	±	14 ^b
1-Methylphenanthrene ^{d,e,f,i}	73.2	±	5.9 ^b
Fluoranthene ^{d,e,f,g,h,i}	651	±	50 ^b
Pyrene ^{d,e,f,g,h,i}	581	\pm	39 ^b
Benz[a]anthracene ^{d,e,f,g,h,i}	335	\pm	25 ^b
Chrysene ^{f,h}	291	\pm	31 ^b
Triphenylene ^{f,h}	108	\pm	5 ^c
Benzo[b]fluoranthene ^{e,g}	453	\pm	21 ^b
Benzo[k]fluoranthene ^{d,e,f,g}	225	\pm	18 ^b
Benzo[e]pyrene ^{d,e,f,i}	325	\pm	25 ^b
Benzo[a]pyrene ^{d,e,f,h,i}	358	\pm	17 ^b
Perylene ^{d,e,f,h,i}	397	\pm	45 ^b
Benzo[ghi]perylene ^{d,e,f,h,i}	307	\pm	45 ^b
Indeno[1,2,3-cd]pyrene ^{d,e,f,h,i}	341	\pm	57 ^b
Dibenz[a,j]anthracene ^{d,e,f,h}	48.9	\pm	4.6 ^b
Dibenz[a,c]anthracene e,h	36.7	\pm	5.2 ^b
Dibenz[a,h]anthracene ^{e,h}	53	\pm	10^{b}
Benzo[b]chrysene ^{d,e,f,h}	53	\pm	12 ^b
Picene ^{d,e,f}	46.6	±	4.7 ^b

^a Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

SRM 1941b Page 5 of 14

Certified values are weighted means of the results from two to six analytical methods [21]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO and NIST Guides [2].

The certified value is an unweighted mean of the results from two analytical methods. The uncertainty listed with the value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [22] with a pooled, within-method variance following the ISO and NIST Guides [2].

^d GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

^e GC/MS (II) on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.

GC/MS (III) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

g LC-FL (total) of total PAH fraction after PFE with DCM.

^h LC-FL (isomer) of isomeric PAH fractions after PFE with DCM.

¹⁹⁹⁹ Interlaboratory Comparison Study [13] with between 21 and 29 laboratories submitting data for each PAH.

Table 2. Certified Concentrations for Selected PCB Congeners^a in SRM 1941b

PCB Cong	geners		ons (dr μg/kg	y-mass basis) ^b
PCB 8	(2,4'-Dichlorobiphenyl) ^{e,f,g,h,i}	1.65	±	0.19^{c}
PCB 18	(2,2',5-Trichlorobiphenyl) ^{e,f,g,h,i}	2.39	<u>±</u>	0.29^{c}
PCB 28	(2,4,4'-Trichlorobiphenyl) ^{e,f,g,h,i}	4.52	±	0.57^{c}
PCB 31	(2,4',5-Trichlorobiphenyl) ^{e,g,h}	3.18	\pm	0.41°
PCB 44	(2,2'3,5'-Tetrachlorobiphenyl) ^{e,f,g,h,i}	3.85	\pm	0.20^{d}
PCB 49	(2,2'4,5'-Tetrachlorobiphenyl) ^{e,f,g,h}	4.34	\pm	0.28^{d}
PCB 52	(2,2',5,5'-Tetrachlorobiphenyl) ^{e,f,g,h,i}	5.24	\pm	0.28^{d}
PCB 66	(2,3',4,4'-Tetrachlorobiphenyl) ^{e,g,h,i,j}	4.96	\pm	0.53^{d}
PCB 87	(2,2',3,4,5'-Pentachlorobiphenyl) ^{e,f,h,j}	1.14	\pm	0.16^{c}
PCB 95	(2,2',3,5',6-Pentachlorobiphenyl) ^{e,g,h,i}	3.93	\pm	0.62^{d}
PCB 99	(2,2',4,4',5-Pentachlorobiphenyl) ^{e,f,g,h,J}	2.90	\pm	0.36^{d}
PCB 101	(2,2',4,5,5'-Pentachlorobiphenyl) ^{e,g,h,i,j}	5.11	\pm	0.34^{d}
PCB 105	(2,3,3',4,4'-Pentachlorobiphenyl) ^{e,f,g,h,i,j}	1.43	\pm	0.10^{d}
PCB 110		4.62	\pm	0.36^{d}
PCB 118	(2,3',4,4',5-Pentachlorobiphenyl) ^{e,f,g,h,i,j}	4.23	\pm	0.19 ^d
PCB 128	(2,2',3,3',4,4'-Hexachlorobiphenyl) ^{e,f,g,h,i,j}	0.696	<u>±</u>	0.044 ^d
PCB 138	(2,2',3,4,4',5'-Hexachlorobiphenyl) ^{e,g,h,j}	3.60	±,	0.28^{d}
PCB 149	(2,2',3,4',5',6-Hexachlorobiphenyl) ^{e,f,g,j}	4.35	\pm	0.26°
PCB 153	(2,2',4,4',5,5'-Hexachlorobiphenyl) ^{e,f,g,h,i,j}	5.47	±,	0.32^{d}
PCB 156		0.507	\pm	0.090^{c}
PCB 170		1.35	\pm	0.09^{d}
PCB 180	(2,2',3,4,4',5,5'-Heptachlorobiphenyl) ^{e,f,g,h,i,j}	3.24	\pm	0.51 ^d
PCB 183	(2,2',3,4,4',5',6-Heptachlorobiphenyl) ^{e,f,g,j}	0.979	\pm	0.087^{c}
PCB 187		2.17	\pm	0.22^{d}
PCB 194	(2,2',3,3',4,4',5,5'-Octachlorobiphenyl) ^{e,f,g,j}	1.04	\pm	0.06°
PCB 195	(2,2',3,3',4,4',5,6-Octachlorbiphenyl) ^{e,g,i,j}	0.645	\pm	0.060^{d}
PCB 201		0.777	\pm	0.034^{c}
PCB 206	(2,2',3,3',4,4',5,5',6-Nonachlorobiphenyl) ^{e,g,h,i,j}	2.42	\pm	0.19 ^d
PCB 209	Decachlorobiphenyle,f,g,h,i,j	4.86	±	0.45^{d}

^a PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [18] and later revised by Schulte and Malisch [19] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, only PCB 201 and PCB 107 (see Table 5) are different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 201 is listed as PCB 200 and the IUPAC PCB 107 is listed as PCB 108.

b Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

- ^e GC/MS (IA) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.
- ^f GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.
- ^g GC-ECD (IB) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (IA).
- ^h GC/MS (II) on a relatively non-polar proprietary phase after Soxhlet extraction with DCM.
- ¹ 1999 Interlaboratory Comparison Study [13] with between 13 and 31 laboratories submitting data for each PCB congener.
- GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).

SRM 1941b Page 6 of 14

Certified values are unweighted means of the results from three to five analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [22] with a pooled, within-method variance following the ISO and NIST Guides [2].

d Certified values are weighted means of the results from three to six analytical methods [21]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO and NIST Guides [2].

Table 3. Certified Concentrations for Selected Chlorinated Pesticides in SRM 1941b

Chlorinated Pesticides	Mass Fractions (dry-mass basis) ^a μg/kg		
Hexachlorobenzene ^{d,e,f,g}	5.83 ± 0.38^{b}		
<i>cis</i> -Chlordane ^{d,e,f,g,h}	0.85 ± 0.11^{c}		
trans-Chlordane ^{d,e,g}	0.566 ± 0.093^{b}		
cis-Nonachlor ^{d,g,h}	$0.378 \pm 0.053^{\circ}$		
trans-Nonachlor ^{d,e,f,g,h}	0.438 ± 0.073^{b}		
$4,4$ '-DDE d,f,g,h	3.22 ± 0.28^{c}		
4.4 '-DDD d,f,g,h	4.66 ± 0.46^{c}		

- ^a Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.
- Certified values are unweighted means of the results from three to five analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [22] with a pooled, within-method variance following the ISO and NIST Guides [2].
- ^c Certified values are weighted means of the results from three to five analytical methods [21]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO and NIST Guides [2].
- d GC/MS (IA) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.
- ^e GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).
- ^f GC/MS (II) on a relatively non-polar proprietary phase after Soxhlet extraction with DCM.
- ^g 1999 Interlaboratory Comparison Study [13] with between 13 and 31 laboratories submitting data for each pesticide.
- ^h GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

SRM 1941b Page 7 of 14

Table 4. Reference Concentrations for Selected PAHs in SRM 1941b

PAHs	Mass Fractions (dry-mass basis) ^a μg/kg		
	1	ıg/K	5
1-Methylnaphthalene ^{d,e,f,i}	127	±	14 ^b
2-Methylnaphthalene ^{d,e,f,i}	276	\pm	53 ^b
2,6-Dimethylnaphthalene ^{d,e,f,i}	75.9	\pm	4.5 ^b
2,3,5-Trimethylnaphthalene ^{d,e,f,i}	25.5	\pm	5.1 ^b
Biphenyl ^{d,e,f,i}	74.0	\pm	8.0^{b}
Acenaphthylene ^{d,e,f,i}	53.3	\pm	6.4 ^b
Acenaphthene ^{d,e,f,i}	38.4	\pm	5.2 ^b
9-Methylphenanthrene ^e	63.5	\pm	2.5°
4-Methylphenanthrene and	80.1	\pm	4.8 ^b
9-Methylphenanthrene ^{d,f}			
2-Methylanthracene ^{e,f}	36	\pm	15 ^b
8-Methylfluoranthene ^d	49.5	\pm	2.7 °
7-Methylfluoranthene ^d	45.4	\pm	1.5 °
1-Methylfluoranthene ^d	42.4	\pm	2.1 °
3-Methylfluoranthene ^d	28.8	\pm	1.3 °
2-Methylpyrene ^d	78.7	\pm	4.0 °
4-Methylpyrene ^d	66.4	\pm	2.6 °
1-Methylpyrene ^d	52.5	\pm	2.3 °
Acephenanthrene ^f	30.5	\pm	1.9°
Benzo[c]phenanthrene ^{d,e,f}	58	\pm	15 ^b
Benzo[a]fluoranthene ^{d,e,f}	73	\pm	18 ^b
Benzo[<i>j</i>]fluoranthene ^e	217	\pm	5°
Indeno[1,2,3-cd]fluoranthene ^f	9.63	\pm	0.34 ^c
Pentaphene ^f	25.3	\pm	1.0°

^a Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

- d GC/MS (I) on 5 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.
- ^e GC/MS (II) on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM.
- f GC/MS (III) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.
- ^g LC-FL (total) of total PAH fraction after PFE with DCM.
- ^h LC-FL (isomer) of isomeric PAH fractions after PFE with DCM.
- ¹ 1999 Interlaboratory Comparison Study [13] with between 14 and 26 laboratories submitting data for each PAH.

SRM 1941b Page 8 of 14

Reference values are weighted means of the results from two to four analytical methods [21]. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance following the ISO and NIST Guides [2].

Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is intended to represent, at the level of one standard deviation, the combined standard uncertainty calculated according to the ISO and NIST Guides [2]. The coverage factor, k, is determined from the Student's t-distribution for the appropriate degrees of freedom and 95 % confidence for each analyte.

Table 5. Reference Concentrations for Selected PAHs of Molecular Weight 300 and 302 in SRM 1941b

PAHs	Mass Fractions (dry-mass basis) ^{a,b,c} μg/kg		
Coronene	72.6	±	4.7
Dibenzo[b,e]fluoranthene	10.3	\pm	0.3
Naphtho[1,2- <i>b</i>]fluoranthene	91.0	\pm	3.1
Naphtho[1,2- <i>k</i>]fluoranthene			
and Naphtho[2,3-j]fluoranthene	79.8	\pm	2.5
Naphtho[2,3-b]fluoranthene	23.5	\pm	0.3
Dibenzo[b,k]fluoranthene	95.6	\pm	3.1
Dibenzo[<i>a</i> , <i>k</i>]fluoranthene	26.6	\pm	0.4
Dibenzo[<i>j</i> , <i>l</i>]fluoranthene	63.8	\pm	1.8
Dibenzo[a,l]pyrene	11.1	\pm	1.0
Naphtho[2,3-k]fluoranthene	10.7	\pm	0.6
Naphtho[1,2-a]pyrene	16.7	\pm	1.4
Naphtho[2,3-e]pyrene	33.2	\pm	2.3
Dibenzo[a,e]pyrene	76.1	\pm	3.6
Naphtho[2,1-a]pyrene	59.2	\pm	1.8
Dibenzo[e,i]pyrene	35.0	\pm	2.4
Naphtho[2,3-a]pyrene	16.5	\pm	0.6
Benzo[b]perylene	38.2	\pm	1.2
Dibenzo[a,i]pyrene	25.5	\pm	1.0
Dibenzo $[a,h]$ pyrene	6.94	±	0.29

^a Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

SRM 1941b Page 9 of 14

Reference values are the means of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's t-distribution for the associated degrees of freedom and 95 % confidence level for each analyte.

^c GC/MS on 50 % phenyl-substituted methylpolysiloxane phase after PFE with DCM [14].

Table 6. Reference Concentrations for Selected PCB Congeners^a in SRM 1941b

Mass Fractions (dry-mass basis)^{b,c} **PCB** Congeners μg/kg (2,2',3,6-Tetrachlorobiphenyl)^{d,e} PCB 45 0.73 0.12 (2,3,3',4-Tetrachlorobiphenyl)^{d,f,g} PCB 56 1.21 0.11 (2,3,4',5-Tetrachlorobiphenyl)^{e,f,g} PCB 63 0.040 0.213 PCB 70 (2,3',4',5-Tetrachlorobiphenyl)^{e,f,g} 4.99 0.29 PCB 74 (2,4,4',5-Tetrachlorobiphenyl)^{e,f,g} 2.04 + 0.15 PCB 77 (3,3',4,4'-Tetrachlorobiphenyl)^h 0.31 0.03 PCB 107 (2,3,3',4,5'-Pentachlorobiphenyl)^{d,e,f,g} 0.628 \pm 0.028 PCB 132 (2,2',3,3',4,6'-Hexachlorobiphenyl)^{d,f,g} 1.28 0.27 \pm PCB 146 (2,2',3,4',5,5'-Hexachlorobiphenyl)^{e,f,g} 1.22 \pm 0.12 PCB 158 (2,3,3',4,4',6-Hexachlorobiphenyl)^{d,e,f,g} 0.65 \pm 0.15 PCB 163 (2,3,3',4',5,6-Hexachlorobiphenyl)^{e,f,g} 1.28 \pm 0.06 PCB 174 (2,2',3,3',4,5,6'-Heptachlorobiphenyl)^{d,e,f,g} 1.51 0.39 PCB 193 (2,3,3',4',5,5',6-Heptachlorobiphenyl)^{d,e,f,g} 0.292 0.075

SRM 1941b Page 10 of 14

^a PCB congeners are numbered according to the scheme proposed by Ballschmiter and Zell [18] and later revised by Schulte and Malisch [19] to conform with IUPAC rules; for the specific congeners mentioned in this SRM, only PCB 201 (see Table 2) and PCB 107 are different in the numbering systems. Under the Ballschmiter and Zell numbering system, the IUPAC PCB 201 is listed as PCB 200 and the IUPAC PCB 107 is listed as PCB 108.

b Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

For these PCB congeners except PCB 77, the reference values are unweighted means of the results from two to four analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [22] with a pooled within-method variance following the ISO and NIST Guides [2]. For PCB 77, the reference value is the mean of results obtained by NIST using one analytical technique. The expanded uncertainty, U, is calculated as $U = ku_c$, where u_c is one standard deviation of the analyte mean, and the coverage factor, k, is determined from the Student's t-distribution corresponding to two degrees of freedom and 95 % confidence level for PCB 77.

^d GC-ECD (IA) on 5 % phenyl-substituted methylpolysiloxane phase after PFE extraction with DCM.

^e GC-ECD (IB) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (IA).

GC/MS (IA) on a relatively non-polar proprietary phase after Soxhlet extraction with 50 % hexane/50 % acetone mixture.

^g GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).

GC/MS NICI on a 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC-ECD (I) fractionated using a PYE column.

Table 7. Reference Concentrations for Selected Chlorinated Pesticides in SRM 1941b

Chlorinated Pesticides	Mass Fractions (dry-mass basis) ^{a,b}
	μg/kg
2,4'-DDE ^{c,d}	0.38 ± 0.12
4,4'-DDT ^{e,f}	1.12 ± 0.42

^a Concentrations reported on dry-mass basis; material as received contains approximately 2.4 % moisture.

Table 8. Reference Value for Total Organic Carbon in SRM 1941b

Total Organic Carbon (TOC) 2.99 % ± 0.24 % mass fraction^{a,b}

Table 9. Information Values for Carbon, Hydrogen, and Nitrogen in SRM 1941b

Elements	Mass Fractions (dry-mass basis) ^a
	%
Carbon	3.3
Hydrogen	1.2
Nitrogen	< 0.5

^a Concentration is reported on a dry-mass basis; material as received contains approximately 2.4 % moisture.

SRM 1941b Page 11 of 14

The reference values are unweighted means of the results from two analytical methods. The uncertainty listed with each value is an expanded uncertainty about the mean, with coverage factor 2, calculated by combining a between-method variance [22] with a pooled, within-method variance following the ISO and NIST Guides [2].

^c GC/MS (IB) on 5 % phenyl-substituted methylpolysiloxane phase; same extracts analyzed as in GC/MS (IA).

^d GC-ECD (IB) on a relatively non-polar proprietary phase; same extracts analyzed as in GC-ECD (IA).

^e GC/MS (II) on a relatively non-polar proprietary phase after Soxhlet extraction with DCM.

f 1999 Interlaboratory Comparison Study [13] with 10 laboratories submitting data for 4,4'-DDT.

^a Concentration is reported on a dry-mass basis; material as received contains approximately 2.4 % moisture.

The reference value for total organic carbon is a weighted mean value from routine measurements made by two laboratories [21]. The uncertainty listed is an expanded uncertainty about the mean, with coverage factor 2 (approximately 95 % confidence), calculated by combining a between-method variance incorporating inter-method bias with a pooled within-method variance. The reporting follows the ISO and NIST Guides [2].

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SRM 1941b Page 12 of 14

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Certificate Revision History: 16 August 2004 (This revision removes the reference values for the butyl tins and makes editorial changes); 15 July 2002 (Original certificate date).

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SRM 1941b Page 13 of 14

APPENDIX A

The laboratories listed below performed measurements that contributed to the certification of SRM 1941b, Organics in Marine Sediment.

Arthur D. Little, Inc; Cambridge, MA, USA Axys Analytical Services; Sidney, BC, Canada B & B Laboratories; College Station, TX, USA Battelle Ocean Sciences; Duxbury, MA, USA

Bedford Institute of Oceanography; Dartmouth, NS, Canada

California Department of Fish and Game; Rancho Cordova, CA, USA

Central Contra Costa Sanitary District; Martinez, CA, USA Chesapeake Biological Laboratory; Solomons, MD, USA

Centro de Investigacionies Energeticas Medioambientales y Tecnologicas; Madrid, Spain

City of Los Angeles Environmental Monitoring Division; Playa del Rey, CA, USA

City of San Jose Environmental Services Department; San Jose, CA, USA

Columbia Analytical Services; Kelso, WA, USA

East Bay Municipal Utility District; Oakland, CA, USA

Florida Department of Environmental Protection; Tallahassee, FL, USA

Manchester Environmental Laboratory; Port Orchard, WA, USA

Murray State University; Murray, KY, USA

Massachusetts Water Resources Authority Central Lab; Winthrop, MA, USA

National Research Council of Canada; Ottawa, Ontario, Canada

National Oceanic and Atmospheric Association (NOAA), National Marine Fisheries Service (NMFS), Auke Bay Laboratory; Juneau, AK, USA

NOAA, National Ocean Service/Center for Coastal Environmental Health and Biomolecular Research; Charleston, SC, USA

NOAA, NMFS, Sandy Hook Marine Laboratory; Highlands, NJ, USA

NOAA, NMFS, Northwest Fisheries Science Center; Seattle, WA, USA

Orange County Sanitation District; Fountain Valley, CA, USA

Philip Analytical Services; Burlington, Ontario, Canada

Serv de Hidrografia Naval; Buenos Aires, Argentina

Skidaway Institute of Technology; Savannah, GA, USA

Southwest Laboratory of Oklahoma; Broken Arrow, OK, USA

Severn Trent Knoxville Laboratory; Knoxville, TN, USA

Texas A&M University, Geochemical and Environmental Research Group; College Station, TX, USA

Texas Parks and Wildlife Department; San Marcos, TX, USA

University of California at Los Angeles, Institute of Geophysics and Planetary Physics; Los Angeles, CA, USA

University of Connecticut, Environmental Research Institute; Storrs, CT, USA

University of Rhode Island, Graduate School of Oceanography; Narragansett, RI, USA

US Department of Agriculture, Environmental Chemistry Laboratory; Beltsville, MD, USA

US Environmental Protection Agency, Atlantic Ecology Division; Narragansett, RI, USA

US Geological Survey, National Water Quality Laboratory; Denver, CO, USA

Woods Hole Group Environmental Lab; Raynham, MA, USA

Wright State University; Dayton, OH, USA

SRM 1941b Page 14 of 14